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Report No. 15960

THE DOW CHEMICAL COMPANY
METALLURGICAL LABORATORIES
MIDLAND, MICHIGAN

FINAL REPORT

Contract Number N9-onr-85900
Task Order 85902

PLASTICITY OF MAGNESIUM ALLOY SINGLE CRYSTALS AND ORIGIN OF SECONDARY MAXIMA (002) POLE FIGURES FOR ROLLED MAGNESIUM ALLOYS

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PLASTICITY OF MAGNESIUM ALLOY SINGLE CRYSTALS AND ORIGIN OF SECONDARY MAXIMA (002) POLE FIGURES FOR ROLLED MAGNESIUM ALLOYS

INTRODUCTION

Part of the previous work under this contract on the effect of temperature on the lattice parameters of magnesium alloys has been published in the Transactions, AIME, 194, (1952), page 207. Additional work on the behavior of single crystals in a polycrystalline aggegate disclosed a complexity of deformation that was too great for analysis based upon the present knowledge of single crystal deformation mechanisms. An attempt to correlate the micro hardness of Mg solid solutions with atomic or ionic radii encountered similar difficulties of analysis because the deformation processes during indentation hardness measurements are not known. It was evident from these studies that it would be necessary to obtain more basic information on plastic deformation.

During deformation a grain in polycrystalline material deforms in a non-homogeneous manner which results from the interaction of adjacent grains. Thus, it is of primary importance to determine the various deformation mechanisms which allow plastic flow under complex stresses. Part of this report describes an attempt to obtain information on this problem by deforming Mg single crystals to various amounts of cold rolling and subjecting single crystal wafers to compressive stresses parallel to the basal plane.

A basic measurement of the contribution of solute elements to solid solution strengthening would be the critical resolved shear stress of alloy single crystals since this quantity would be independent of orientation and would be relatively free of effects produced by uncontrolled metallurgical variables. By proper choice of the alloying elements, the relative effects of lattice distortion and electron/atom ratios might be evaluated. The results of Mg-Zn and Mg-In alloy crystals are presented in this report.

Because part of the objective of this investigation was concerned with plastic deformation mechanisms which could be extended ultimately to an explanation of the behavior of alloyed polycrystalline material, the experimental program was expanded to include a study of the role which Ca occupies during the rolling of FS sheet. Previously, Ca has been reported to be instrumental in producing a double maxima in the basal pole figure of Mg sheet but the mechanism was unknown. One part of this report is therefore devoted to an explanation of the origin of the double maxima in the basal pole figure of Mg sheet.

TABLE OF CONTENTS

ABSTRACT

The "double peak" in the (002) texture of FS alloy results from the overlapping of two displaced single peaks which are independent of the presence of Ca. The angle of displacement and sense of rotation are related to the frictional forces of the top and bottom roll and are a function of the roll diameter and per cent reduction per pass respectively.

The mechanisms of plastic deformation during the rolling of single crystals are (002) slip, {102} twinning, slip on the reoriented (002) plane, bend plane formation and {101} slip (?) in highly constrained areas.

Compression stresses parallel to the basal plane in crystal wafers demonstrated that {101} slip will only occur with great difficulty at room temperature. Twinning is always accompanied by an accomodation plane and the interaction of accomodation planes inhibit twin growth. Twins may increase laterally from one side when constraints are present. Mg may be de-twinned by the application of a reverse stress as well as twinned by residual microstresses. A discontinuity is present after the de-twinning occurred.

Multiple nucleation of the cast alloy rods increased rapidly with increasing solute. The critical resolved shearing stress for Mg-Zn single crystals increased linearly with atomic per cent Zn to 0.32 atomic per cent Zn. The addition of 0.09 atomic per cent In increased the resolved shear stress of pure Mg from 46 gm/mm^2 to 56 gm/mm^2.

PART I

ORIGIN OF THE DOUBLE MAXIMA IN THE (002) POLE FIGURE OF ROLLED MAGNESIUM

INTRODUCTION

The rolling texture of Mg is generally represented as one in which the basal planes lie near the rolling plane with a greater spread in the rolling direction than in the transverse direction.[(1),(2),(3)]. The formation of a double maximum in the basal plane texture may be due to {101} twinning caused by Ca additions[(4),(5)] although Hargreaves[(6)] found the double maximum without Ca.

The existence of a preferred crystallographic direction in the rolling direction for Mg is doubtful since it has been reported as [100] and [210],[(7),(8)].

SUMMARY OF EXPERIMENTAL RESULTS

1. FS-1 and FS-W alloy sheet exhibit displaced basal plane maxima which lie in a plane containing the R.P. normal and the R.D.

2. The surface orientation consisted of a single peak, either displaced or not depending upon the rolling technique. The interior of the sheet exhibited a double maximum which resulted from the overlapping of two single displaced peaks.

3. The angle of displacement between the R.P. normal and the basal plane maxima varied from 10-15° on the 8" mill to 5° on the 3" mill.

4. The sense of the angle of displacement of the basal plane maxima with respect to the R.D. changed with per cent reduction per pass for both alloys.

5. No evidence of a preferred crystallographic direction

coincident with the R.D. was observed.

CONCLUSIONS

1. The presence of Ca is not necessary for the formation of double maxima in the (002) rolling texture of FS alloy.

2. The double maxima results from the overlapping of two single displaced peaks, each displaced peak being related to the top or bottom roll.

3. The displacement and sense of rotation of the (002) maxima from the R.P. normal results from the effect of frictional forces on the plastic flow during rolling rather than a new crystallographic slip mechanism caused by Ca.

4. The (002) planes are randomly oriented about the [001].

EXPERIMENTAL PROCEDURE

Two lots of FS alloy with the following composition* were used in these experiments:

	Al	Zn	Mn	Ca	Cu	Fe	Ni
FS-W	2.90	0.96	0.51	0.023	0.01	0.002	0.0006
FS-1	2.60	0.97	0.45	0.142	0.002	0.001	0.0004

*Weight per cent.

Slabs were removed from the cast billets, scalped 5/16", machined into rectangular blocks and soaked 16 hours at 800F in an SO_2 atmosphere prior to hot rolling. The blocks were hot rolled to various thicknesses at temperatures between 500 and 700F in order that a constant thickness of 0.064" would be obtained after different increments of cold rolling. The material was rolled by two procedures - with and without end removal between successive passes. The reduction in thickness per pass during cold rolling was 0.001" - 0.002" on an 8" mill at 100 feet/minute.

The orientation of the basal planes was determined with an x-ray spectrometer, using a surface reflection technique(9). Supplemental information on the alignment of a crystallographic direction with the rolling direction was obtained with a conventional transmission film technique using Ni filtered Cu radiation.

RESULTS

Figure (1) shows the variation in the distribution of the basal planes in the surface layer of FS-1 alloy after 30% reduction by cold rolling. This graph represents the intersection of a plane containing the R.P. normal and the R.D. with the surface defined as a pole figure. The as rolled surface exhibits a peak intensity which is displaced 12 degrees from the R.P. normal and rotated away from the R.D. [Hargreaves(6) has demonstrated that the peak of the intensity lies in a plane containing the R.P. normal and the R.D.]

After etching the specimen to the depths noted on the graph, further orientation measurements disclosed that the intensity decreased as the depth below the surface increased. Simultaneously, the intensity related to the bottom roll steadily increased. As the midpoint of the sheet thickness was passed, the intensity related to the bottom roll exceeded the corresponding intensity of the top roll. The intensity of the bottom surface became nearly equal to the intensity of the top surface and showed that the texture consisted of two displaced peaks which were mirror images across the midpoint of the sheet.

Alternate end reversal between passes produced an (002) peak intensity which coincided with R.P. normal while the lower layers exhibited a double maxima which was displaced from the R.P. normal, (Figure 2).

Figure (3) represents the orientation of FS-W processed in a similar manner to Figure (1). The development and striking resemblance to the texture found in FS-1 is evident.

Consideration of the development of the displaced basal peaks and their relationship to the rolling direction suggested that friction between the sheet and the rolls was a possible cause of the displacement. Since the material had been prepared by very light reductions per pass, additional work was performed by varying the reduction per pass to the limit of cracking.

The effect of reduction per pass upon the angle of peak displacement was performed with a 3" mill with the results shown in Figure (4). The reversal in the angle of tilt from the R.P. normal is seen to be a function of the reduction per pass, the transition region being defined between 3.75 and 9 per cent reduction/pass. Furthermore, the amount and reversal of the displacement angle was independent of Ca. Besides attaining a constant angle of displacement, it will be noted that the displaced peak occurs at 5° as compared to 10-15° on the 8" mill. Apparently, the angle of departure from the R.P. normal is a function of the roll diameter as well as reduction per pass.

<u>Presence of Preferred Crystallographic Direction in Rolling Direction.</u>

Transmission photograms were made of both alloys to determine whether a specific crystallographic direction coincided with the R.D.; the $\{100\}$ and $\{101\}$ reflections were used as the criterion. After etching a specimen of FS-1 cold rolled 25% from one side until a surface layer, 0.003" thick, remained, a transmission photogram was made with the x-ray beam perpendicular to the R.P. with the R.D. vertical, Figure (5). The $\{100\}$ and $\{101\}$ reflections have a uniform, diffuse density with no evidence of preferred directionality of alignment with respect to the R.D. Intermediate layers of the sheet were obtained by etching and again the $\{100\}$ and $\{101\}$ rings were uniform.

DISCUSSION OF RESULTS

Hargreaves[6] reported a similar study of orientations in AM503 (M Alloy) and concluded that the [001] was perpendicular to the R.P. in the as rolled surface layers. The central region of the sheet had two peaks whose [001] directions were inclined at 15° to the R.P. normal and lay in a plane containing the R.P. normal and the R.D. No explanation for the displaced peaks was offered except that it was stated that Ca was not necessary for the formation of the displaced peaks.

There is general agreement between Hargreaves findings and the present work although some of the details are conflicting. A thin surface layer of the sheet processed by alternate end reversal between passes had the [001] parallel to the R.P. normal while unidirectional rolling had the [001] tilted away from the R.P. normal. Thus, Hargreaves' results on the [001] being parallel to the R.P. normal in the surface layers probably reflects the rolling technique rather than an intrinsic difference in the deformation mechanism.

It was also reported that the surface layers with a single basal plane peak had the [100] parallel to the R.D. while the interior of the sheet was randomly oriented about the [001][6]. In the present experiment no directionality about the [001] was found at any level within the sheet. However, since a relatively small scatter would destroy the directionality in the basal plane, it is not surprising that the results are contradictory[10].

The surface layers in metals from any forming operation usually exhibit orientations which are not found at the center of the cross-section[11]. The departure of the surface layers from the ideal orientation results primarily from the effect of friction on the metal flow during forming. During rolling there is only one point on the surface of contact between the rolls and the material at which the metal and the roll surface move with the same velocity and this is defined as the "no-slip point"[12]. Between the point of entry of the rolls and the no-slip point, the surface of the material is moving slower than the roll surface and the resulting friction draws the sheet between the rolls. Between the no-slip point and the roll exit, the metal surface moves faster than the roll surface; consequently, the friction opposes the delivery of the sheet.

For light reductions per pass, the surface flow is more rapid than the interior because the frictional forces are additive in the direction of rolling. The shearing stresses within the sheet must be a maximum at the outer surfaces and decrease to a minimum at the center. Because Mg deforms plastically by (002) slip, $\{102\}$ twinning and slip on the reoriented basal plane with $\{101\}$ slip being observed under special stress conditions below 225C, the differential flow resulting from the variation in shearing stresses across the sheet thickness must be accomplished by the former three mechanisms. The experimental evidence for light reductions per pass is in excellent agreement with the friction hypothesis since the peak of the basal planes is rotated away from the rolling direction.

The friction hypothesis was tested further by altering the position of the no-slip point, and consequently, the relative amounts of forward and back friction. The no-slip point may be moved toward the entry side of the rolls by front tension, lubrication or heavy reductions per pass, the latter method being used in these tests.

With heavy reductions per pass, the no-slip point moves toward the entry side increasing the region in which friction retards the forward flow of the surface layers. The surface layers of the sheet become constrained and the flow is less rapid than that occurring in the interior, thus producing a displaced basal plane peak which is tilted <u>toward</u> the R.D. The data presented in Figure (4) demonstrate that the basal planes do reverse the direction of tilting in accordance with the friction hypothesis. These data emphasize the general principle that the direction of flow of the metal is the controlling factor in producing textures[10].

The constancy of the angle of displacement (10-15°) of the basal plane peak from the R.P. normal has caused previous workers to conclude that some additional slip mechanism was responsible. However, the present results show that the angle of displacement may be a function of the roll diameter as well as the per cent reduction per pass and indirectly substantiate the hypothesis that friction forces are the true cause of the secondary maxima in the (002) pole figure of rolled Mg alloys.

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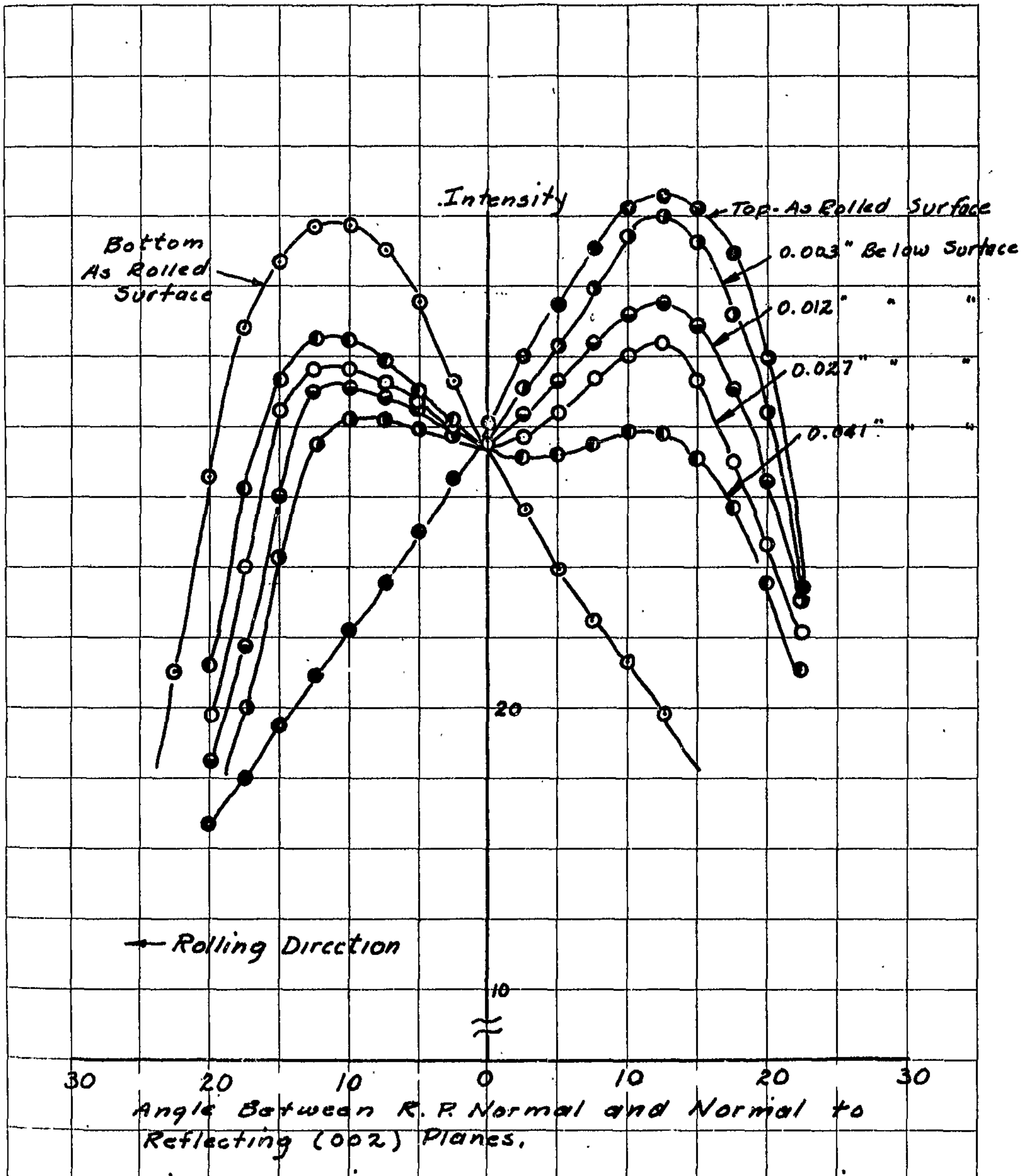

Fig.(1) Variation in Orientation of (002) Planes With Depth Below the As Rolled Surface in FS-1 Alloy. Sheet Rolled in One Direction With 0.001"–0.002"/pass Reduction.

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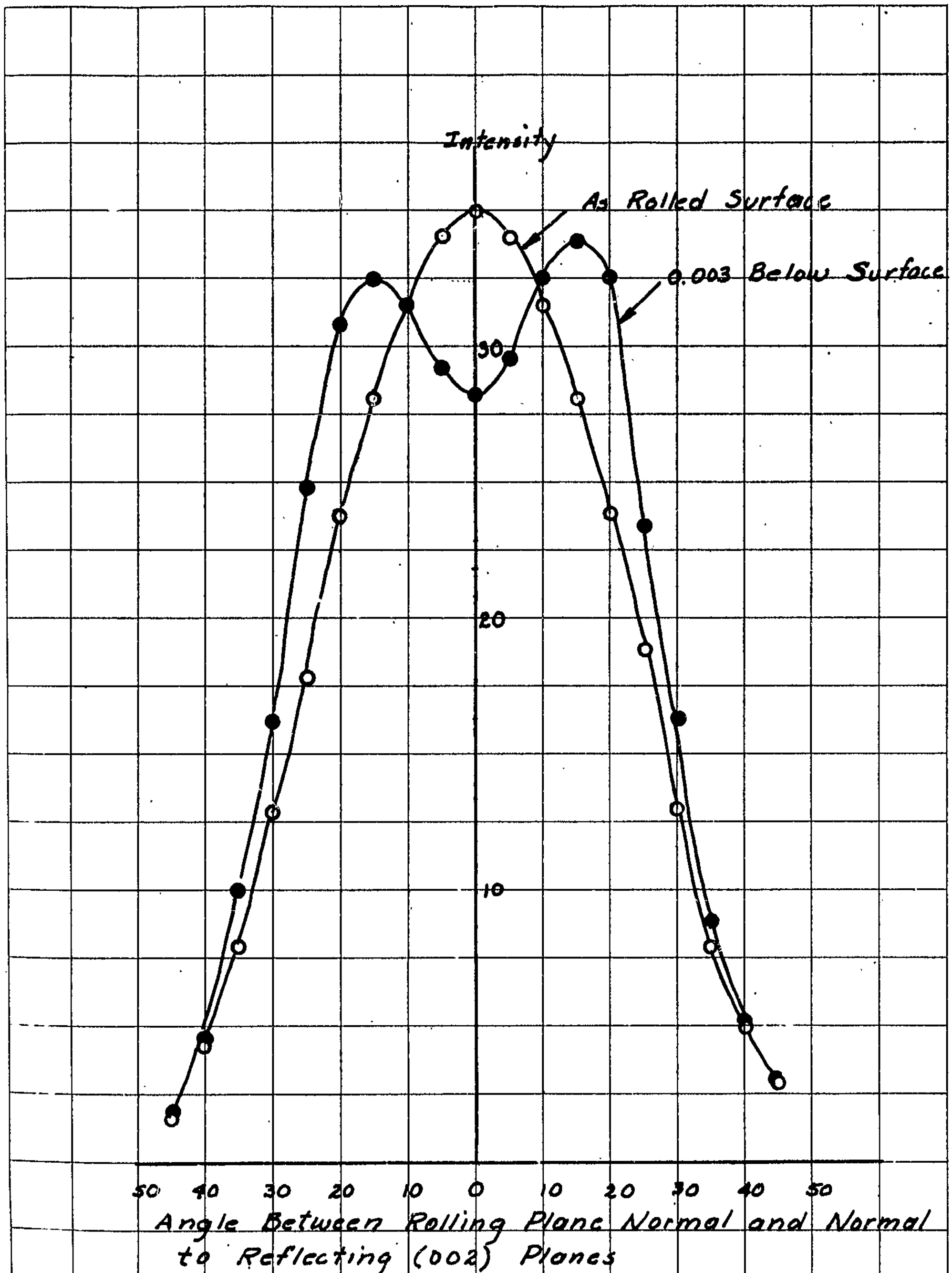

Figure (2)

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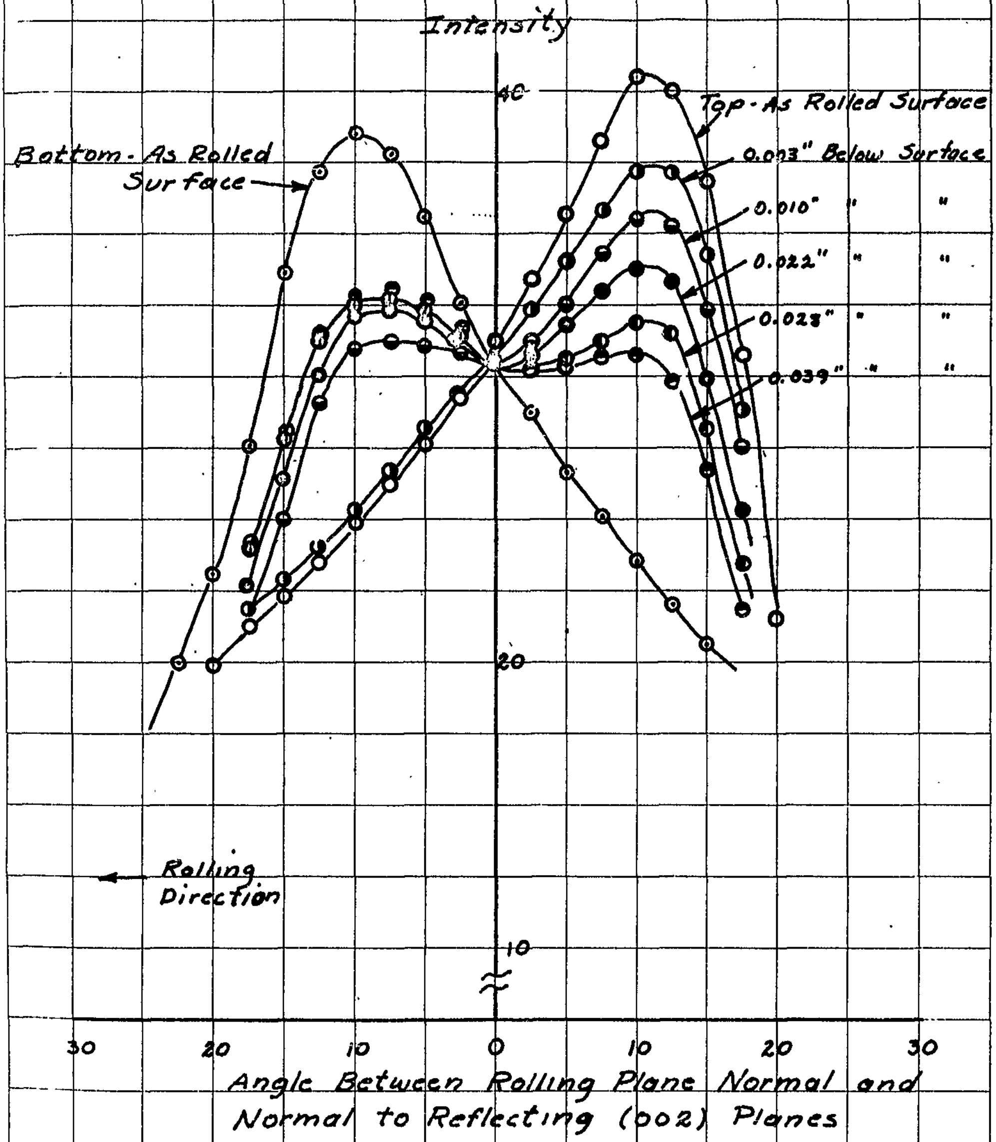

Fig(3) Variation in Orientation of (002) Planes With Depth Below the As Rolled Surface in FS-ω Alloy. Sheet Rolled in One Direction With 0.001"-0.002"/pass Reduction

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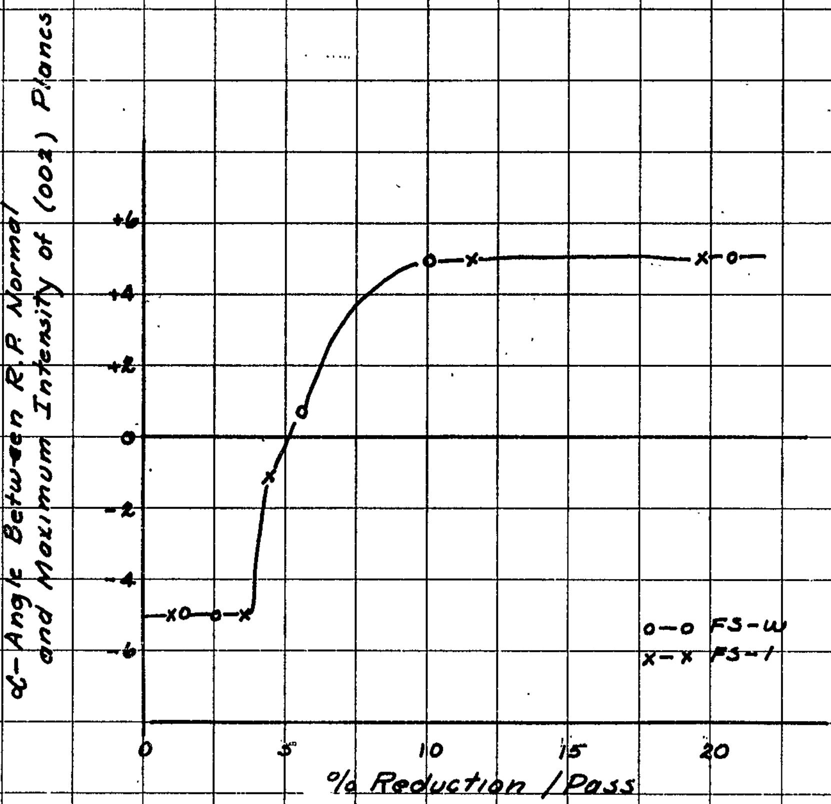
Figure (4)

The Variation in Displacement of the Basal Plane Maximum with the Percent Reduction Per Pass.



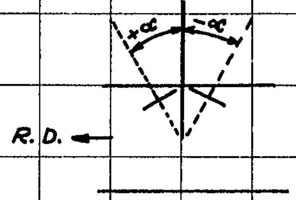


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FIGURE (5)

Negative Number X - 65497

Transmission photogram of 0.003" surface layer of FS-1 sheet.

PART II

SINGLE CRYSTAL ROLLING EXPERIMENTS

SUMMARY OF RESULTS

The mechanisms of plastic deformation during the rolling of single crystals are (002) slip, {102} twinning, slip on the reoriented (002) plane, bend plane formation and a second slip system in a highly constrained region.

EXPERIMENTAL PROCEDURE

In an attempt to elucidate more completely the formation of the double peaks found in the polycrystalline material reported in Part I, square single crystals of Mg, 6" long x 0.5" x 0.5", were subjected to various amounts of cold rolling. The effect of increased deformation was documented with metallographic observation.

RESULTS

The original orientation of a high purity Mg crystal was $X_0 = 52°$, $\lambda_0 = 52°$. After a reduction of 2.76%, basal slip lines were observed on the polished surfaces which were perpendicular to the rolling plane, Figure (1). Further reduction to 5.5% produced more decidedly lamellar regions of slip but did not alter the spacing of the slip lines appreciably, Figure (2). Several large {102} twins were present, Figure (3). Because the specimen progressively bent during rolling, it was necessary to adjust its position on the metallograph stage. Consequently, the slip lines in Figure (2) appear to be a greater angle to the top edge which is the rolling plane than they actually were. Furthermore, opposite polished surfaces were photographed for salient features; hence, the basal slip lines will be often reversed in their slopes in the series of photomicrographs.

The {102} twins which formed in the early stages of reduction were those which were as close to being perpendicular to the rolling plane as the orientation would permit. Further reductions produced such complex twinning that no geometrical relationship could be determined.

After 15.5% reduction, the density of the basal slip lines increased and profuse twinning was observed, Figure (4). Basal slip within the twins and bend planes appear to be the predominating mechanisms with the bend planes being observed at the intersection of the twins.

The crystal deformed in a heterogeneous manner with further cold rolling and several cracks were noted after 35.2% reduction. Severe localized distortion and complex deformation occurred. The density of basal slip lines increased and severe twinning was observed, Figure (5). Marked basal slip on the reoriented twinned material was prevalent as well as 2nd order twinning within the primary twins. In addition, a decided bending of the basal planes was observed. Figure 6 illustrates a severely distorted section in which two distinct families of slip lines are visible as well as twinning. The second set of lines was demonstrated to be slip lines and not fine deformation bands since the lines disappeared after etching. The crystal was so badly distorted that the second set of slip lines could not be identified by x-ray or trace analysis but $\{101\}$ slip would be the most logical assumption.

Two additional square crystals, $X_o = 40°$, $\lambda_o = 41°$ and $X_o = 32°$, $\lambda_o = 32°$ were subjected to the same rolling procedure with similar results.

DISCUSSION OF RESULTS

The mechanisms of plastic deformation which are operative during rolling are the same as those obtained during the tensile extension of Mg single crystals:

(a) (002) slip.
(b) $\{102\}$ twinning.
(c) Slip on the twinned (002)

(d) Bend plane formation
(e) $\{101\}$ slip (?) in locally constrained areas.

The observation of a second slip system in a localized highly strained region indirectly substantiates the finding that $\{101\}$ slip may occur at room temperature when the basal plane is restrained severely[1]. It would seem reasonable to conclude that $\{101\}$ slip cannot play a major role in the deformation of Mg at room temperature since continued rolling caused fracture rather than increased $\{101\}$ activity.

The sequence of operation of the mechanisms observed during rolling illustrates the interplay of $\{110\}$ bend planes to strain hardening, twinning and fracture. When sufficient deformation has occurred on the (002) and the reoriented (002) planes within the twins, a decided increase in the number of bend planes was observed. Thus, bend planes may occupy an analogous position to the occurrence of cross slip in the f.c.c. system with respect to their contribution to strain hardening. Because twinning may relieve the bending stresses produced by basal plane flexure, there will be an intimate association of twinning and bend planes. Ultimately, fracture occurs when the mechanisms interlock and lead to a highly strained state in which further uniform extension is prevented.

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FIGURE (1)

Negative No. 32058 50X

Basal slip after 2.76% reduction.

FIGURE (2)

Negative No. 32059 50X

Basal slip after 5.5% reduction.

FIGURE (3)

Negative No. 32060 50X

Large {102} twins after 5.5% reduction in isolated regions.

FIGURE (4)

Negative No. 32062 50X

Basal slip, {102} twinning, bend planes and increased slip within twins after 15.5% reduction.

FIGURE (5)

Negative No. 32061 50X

Profuse twinning, slip within twins and increased bend plane activity after 35.2% reduction.

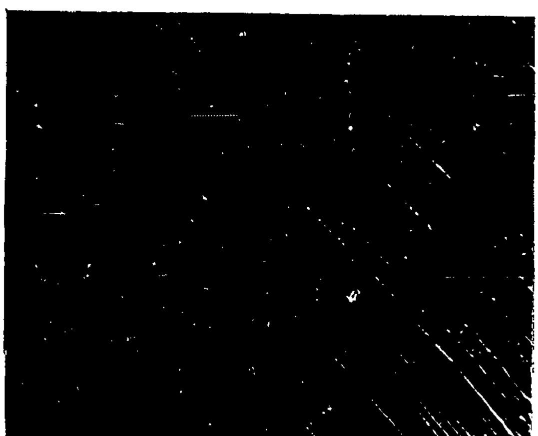

FIGURE (6)

Negative No. 32063 50X

Localized region which contained two slip systems.

PART III

COMPRESSION OF SINGLE CRYSTAL WAFERS

INTRODUCTION

The phenomenon of basal plane bending in the plastic deformati of h.c.p. metals appears to be significant. Jillson[1] suggested that the bending of the basal plane is fundamental to the slip process and the inhomogeneous shearing by slip was simply a consequence. Orowon[2] concluded that bending or kinking assummed equal importance with slip and twinning in Zn although Barrett and Hess[3] believe that kinking is simply a deformation band. Previous work on Mg suggested that basal flexure was intimately related to twinning[4].

The cleavage faces of Zn crystals have been used to study the movement and angle of basal flexure, formation of twins and accomodation planes[1],[5],[6]. Because Mg does not cleave as readily as Zn, no systematic metallographic study of compressed wafers of Mg has been attempted. A technique of specimen preparation, straining and metallographic observations is presented in this section.

SUMMARY OF RESULTS

1. At room temperature slip on the $\{101\}$ plane was not observed when the basal plane was subjected to compressive stress.

2. Twinning is accompanied by accommodation planes and the interaction of accommodation planes inhibit twin growth.

3. Twins may increase in thickness from one side when constraints are present.

4. Mg may be de-twinned by the application of a reverse stress as well as twinned by residual micro stresses.

5. The original lattice is not restored at the de-twinned site as a residual fold persists.

EXPERIMENTAL PROCEDURE

Single crystals of Mg which were oriented by the conventional Greninger method were placed in a tube filled with paraffin and sawed with a jeweler's saw in a miter box arrangement. The crystal sections were subsequently etched in 10% acetic acid and Laue patterns were made after each etching until the reflections were substantially free of asterism. The polished tablets were compressed with Phillip screws in an attachment which fitted the stage of the metallograph.

EXPERIMENTAL RESULTS

Figure (1) is a back reflection Laue pattern of the cut and polished surface prior to compression, the beam being perpendicular to the surface.

Following a small amount of compressive strain, twins were observed to grow rapidly with audible clicking. After the twin had nucleated, the rate of growth was determined by the strain rate; the growth proceeding smoothly without clicking[(1),(6)].

Figures (2) to (7) show the development of two crossed twins. Because $\{102\}$ twins do not form true intersections, a marked increase in stress was necessary to continue the twin across the first one. Frequently, accommodation or bend planes obstructed the longitudinal growth of the twin until sufficient additional stress was supplied to force the twin tip through the discontinuity. The photo micrographs illustrate the complex deformation possible in a highly constrained area with second generation twinning, accommodation planes, reoriented basal slip appearing with the progenitor twin.

The localized blocking of lateral growth or twin thickening by an impinging twin was observed as would be expected. With increased deformation the angle of tilt across the accommodation plane increased and often substructures with curved walls developed. Basal slip on the reoriented twinned material was commonly observed.

The movement of accommodation planes during twinning was recorded in Figures (8) to (11). The stress was applied at a point which corresponds to the right hand edge of the micrograph; the twins were initially nucleated at the point of maximum stress. Distinct accommodation planes were seen parallel to the twins and separated from the twins by a distance equal to 3.5 times the twin thickness. With increased stress, the accommodation plane suddenly reversed its direction and began to approach the twin again; the first twin ceased its growth. Study of the micrographs revealed that a new twin had formed in the lower left corner; consequently, its accommodation plane had the opposite direction. With further stressing, the lower left twin continued to increase while the two families of impinging twins became stabilized from their mutual constraints. The oppositely traveling folds did not annihilate one another since the original basal folds persisted. However, the decreased sharpness of the fold indicated that some interaction had occurred.

The relationship of accommodation folding to twinning is shown in Figures (12) to (14). With the stressing point at the right of the micrograph, Figure (12) the twin furthest away from the stressing screw widened rapidly. The side of this large twin toward the stressing screw remained motionless while the side toward the un-twinned region grew freely. Thus, if a situation arises in which free migration of accommodation planes is inhibited, a twin may widen laterally from one side.

After an original compressive stress sufficient to nucleate several twins, the specimen was unloaded and a new stress parallel to the basal plane but 90° from the original stress was applied. This application of an opposite stress produced de-twinning, Figures (15) to (18). However, a residual discontinuity remained after the twin had disappeared which possessed a small angle of tilting since various oblique lighting arrangements did not emphasize it further. Figure (16) shows a new twin which stopped at this discontinuity, and it is interesting to note that an end accommodation fold formed at the intersection of the twin and the residual fold. Apparently, the residual faulted structure was not properly oriented to relieve the stressed twin tip. The de-twinning of Mg does not produce a completely normal structure since the fold prevented twin propogation at the lower stress. With increased stress, the twin penetrated this barrier and grew rapidly while the amount of folding in the end accommodation plane increased.

An additional type of deformation was observed during compression which have been called mosaic walls[6]. The principle differences between mosaic walls and accommodation folding are: mosaic walls have greater angles of folding and are not confined to a specific plane. In appearance the mosaic walls consist of straight segments and curved portions, Figures (19) to (25). With increasing stress the mosaic walls moved freely and new intersecting loops developed. Upon further compression, some of the intersecting loops combined to increase the amount of folding in the original straight segment although several branches persisted at the terminals. Recombination of some branches occurred with increased strain but no criterion for the absorption selection could be ascertained.

Measurement of the angle between the applied stress and the mosaic wall segment gave an average result of $41° \pm 3$ for 22 measurements indicating that the formation and mobility of these walls must depend upon a shear stress parallel to the wall.

After removal of a pre-compression stress with the specimen remaining under observation, a de-twinning was seen. Since it has been demonstrated that de-twinning may occur by appropriately directed stresses, residual micro-tensile stresses undoubtedly produced it. Because the specimen was viewed at 100 x, the area of observation constituted a small fraction of the total specimen. Therefore, it is probable that a sizeable amount of de-twinning occurred and may be significant.

DISCUSSION OF RESULTS

Doan and Thomasen[7] proposed the hypothesis that residual micro-stresses produced un-twinning in Mg to explain the effect of cyclic stressing upon the tangent elastic modulus. This hypothesis was later used to explain the results of indentation tests of M1 sheet[8]. However, from x-ray examination of compressed sheet, Hess and Dietrich[9] concluded that no significant amount of un-twinning could be produced by residual internal stresses upon load removal.

Because un-twinning by residual stresses was directly observed in the present experiments, it must be concluded the x-ray technique was not sufficiently sensitive. Although no quantitative data on the amount of un-twinning is available, the proof of its existence lends support to the hypothesis of Doan and Thomasen.

The apparent ease of formation and mobility of mosaic walls in Mg suggests that this mode of deformation may assume considerable importance in polycrystalline material. Grains with orientations which are not amenable to basal slip may undergo deformation. The formation of mosaic walls would be especially beneficial in furnishing an additional mechanism for preserving contiguity at grain boundaries since the curved walls appearing in the basal plane must indicate a non-basal slip mechanism.

Attempts to derive a relationship between the observed interval of separation of accommodation planes and the twin with the width of the twin in terms of a maximum possible shear gradient in the material are unlikely. Additional twinning beyond the field of metallographic observation would require accommodation planes with opposite directions of motion which could interact with the first set of accommodation planes and reverse their directions. This would lead to stablized accommodation planes which could be independent of the twin width and shear gradient. However, it has been clearly demonstrated in these tests that twinning will be halted or restricted if the matrix adjacent to the twin cannot undergo folding to alleviate the twinning shear.

An extremely important but indirect conclusion may be deduced from these compression tests. Although compression parallel to the basal planes produced a relatively high resolved shear stress on the $\{101\}$ planes, $\{101\}$ slip was never observed. Thus, additional evidence has been obtained that activating $\{101\}$ slip is extremely difficult to accomplish at room temperature. It is difficult to rationalize this result with the reported $\{101\}$ slip when the basal plane is constrained (4).

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FIGURE (1)

Negative No. X-65518

Back reflection Lane pattern with x-ray beam perpendicular to cut surface.

FIGURE (2)

Negative No. 32578 100X

Development of two crossed twins with increasing stress.

FIGURE (3)

Negative No. 32579 100X

The start of complex deformation at the twin intersection.

FIGURE (4)

Negative No. 32580 100X

Increased twin width with increasing compressive stress.

FIGURE (5)

Negative No. 32581 100X

Severe distortion and second generation twinning at intersection.

FIGURE (6)

Negative No. 32582 100X

Numerous bend planes forming adjacent to twin intersection.

FIGURE (7)

Negative No. 32583 100X

Localized distortion has become sufficiently interlocked that fracture occurred with additional compression.

FIGURE (8)

Negative No. 32522 100X

Movement of accommodation planes during twinning.

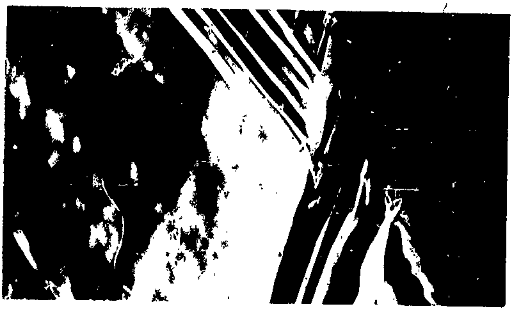


FIGURE (9)

Negative No. 32521 100X

Reversal of direction of motion of accommodation plane with increased stress. Note twin in lower left corner of micrograph.

FIGURE (10)

Negative No. 32519 100X

Twin at lower left edge has grown andaccommodation planes have moved closer to crossed twins. Note that crossed twins have not widened appreciably.

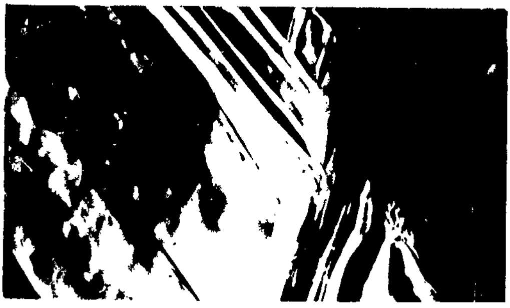


FIGURE (11)

Negative No. 32520 100X

Crossed twins are unable to grow and fine twins are forming.

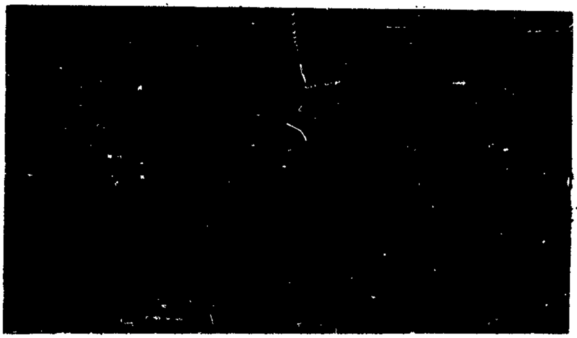


FIGURE (12)

Negative No. 32524 100X

Compressive stress applied horizontally.

FIGURE (13)

Negative No. 32525 100X

The right side of the large twin remains stationary while the left side grows readily. Note lack of growth of smaller twins at right side.

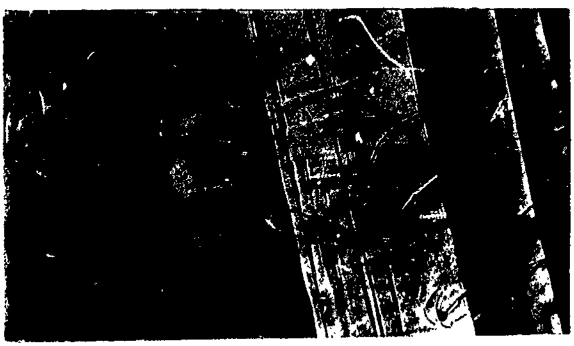


FIGURE (14)

Negative No. 32526 100X

Formation of accommodation plane to left of large center twin.

FIGURE (15)

Negative No. 32464 100X

Intermediate stage in de-twinning upon application of reversed stress. Twins on upper left have started to disappear.

FIGURE (16)

Negative No. 32465 100X

Increased stress has de-twinned material but a residual discontinuity persists. Note that new twin ceases at fold where old twin was present.

FIGURE (17)

Negative No. 32466 100X

New twin has penetrated barrier and is widening smoothly. End accommodation plane has formed.

FIGURE (18)

Negative No. 32467 100X

End accommodation plane has increased folding with new twins being formed in its immediate vicinity.

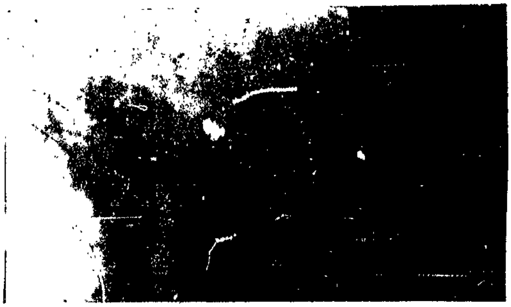


FIGURE (19)

Negative No. 32566   100X

Movement of mosaic wall with increasing stress. Stress applied horizontally.

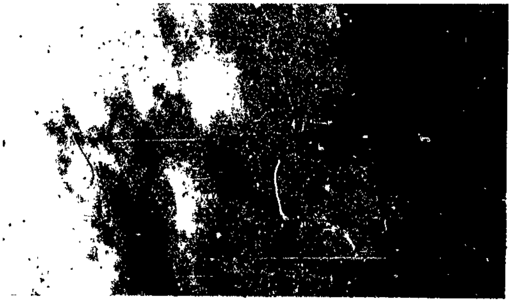


FIGURE (20)

Negative No. 32567   100X

Straight line has become curved.

FIGURE (21)

Negative No. 32568   100X

Several intersecting walls have developed.

FIGURE (22)

Negative No. 32569   100X

Recombination of curved sections into straight line with increased folding dent.

FIGURE (23)
Negative No. 32570 100X
Curvature of wall has increased as well as amount of folding.

FIGURE (24)
Negative No. 32571 100X
More irregular mosaic walls have developed.

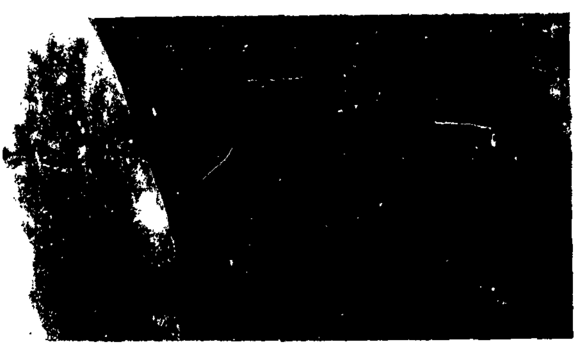


FIGURE (25)
Negative No. 32572 100X
Recombination of the walls has occurred and the step wise process of folding has begun.

PART IV

ALLOY SINGLE CRYSTALS

SUMMARY OF RESULTS

1. Multiple nucleation increased with increasing solute content.

2. Alloys of 0.5% Al could not be converted to single crystals in a graphite mold. The source of nucleation was probably Al_4C_3.

3. The critical resolved shearing stress increased linearly with increasing atomic per cent Zn to 0.32 A.%.

4. The addition of 0.09 A.% In increased the resolved shearing stress of Mg from 46 gm/mm^2 to 56 gm/mm^2.

INTRODUCTION

The contribution of solute elements to solid solution hardening and their effect on the plastic properties is not completely understood. Some of the difficulty is believed to be the result of uncontrolled metallurgical variables such as purity, grain size[1]. This section describes some experimental work with alloy crystals to minimize the variables.

Solid solution hardening has been attributed to lattice strain and the mean free electron to atom ratio from studies on dilute aluminum solid solutions of polycrystalline material[1]. Previous work on alloy Mg crystals is scanty, the only results being those of Schmid and coworkers on Mg-Al and Mg-Zn alloys[2]. The resolved shear stress varied linearly with the atomic per cent solute to 2 atomic per cent Zn and 6 atomic per cent Al, the Zn additions showing a markedly greater increase than Al for the same per cent addition.

EXPERIMENTAL PROCEDURE

Alloys with the compositions listed in Table I were prepared and hot extruded to 0.5" diameter rod. The rods were converted to single crystals, 6" long x 0.5" diameter, in a temperature gradient furnace using an A atmosphere.

Strain measurements were made with SR4 strain gages mounted back to back and the crystals were loaded by a 5/1 lever arm system with a controlled flow of graded Mg powder. An attempt to obtain improved axiality of loading was made by utilizing high strength 0.015" diameter wire seated in semi-spherical seats in the grips as the supports.

The composition of the alloys was selected upon the basis of an appreciable solid solubility in Mg primarily with the effect of the valence of the solute as secondarily important. From this series it was hoped that the effect of lattice distortion could be separated from the contribution of varying electron to atom ratios.

The orientation of the crystals was determined from back reflection photograms with a Greninger net; four exposures at 90° positions were averaged for each value.

RESULTS

(a) Growth of Crystals

It was impossible to grow 0.5% Al crystals in an Acheson graphite crucible under a wide range of cooling rates and temperature gradients because of multiple nucleation, possibly caused by Al_4C_3. Because of the neglible solubility of Mo in Mg, Mo sheet liners were used to insulate the Mg-Al alloy from the crucible. The alloy wet the surface of the sheet during melting and the Mo foil strongly adhered to the crystal. This idea was abandoned after several unsuccessful trials.

The Mg-Li alloys became largely depleted in Li during the lengthy solidification cycle. Consequently; although single crystals were obtained readily, the Li content was reduced to a few hundreths of one per cent and the crystals were useless for this investigation.

Generally, multiple nucleation increased rapidly with alloy content, the 0.32 A.% Zn alloy being converted to a single crystal with difficulty: 7 crystals were obtained from 90 trials. The 0.16 A.% Zn alloy yielded 8 crystals in 21 trials.

The lower In, Hg and Cd compositions had a probability of 1 in 4 that a single crystal would form.

Chemical analyses of crystals from various sections along the rod showed a maximum longitudinal variation of solute of 0.02% over a 6 inch length. Metallographic examination of the Zn alloy disclosed slight evidence of coring, the slow cooling rate allowing sufficient diffusion to minimize micro segregation.

(b) Critical Resolved Shear Stress Determinations.

Table II contains the results of the tensile testing of single crystals of Zn, In and pure Mg. The Hg and Cd crystals yielded such erratic results that further analysis of these data was not attempted. At present no reason is known for the wide variation in resolved shear stress values in these alloys.

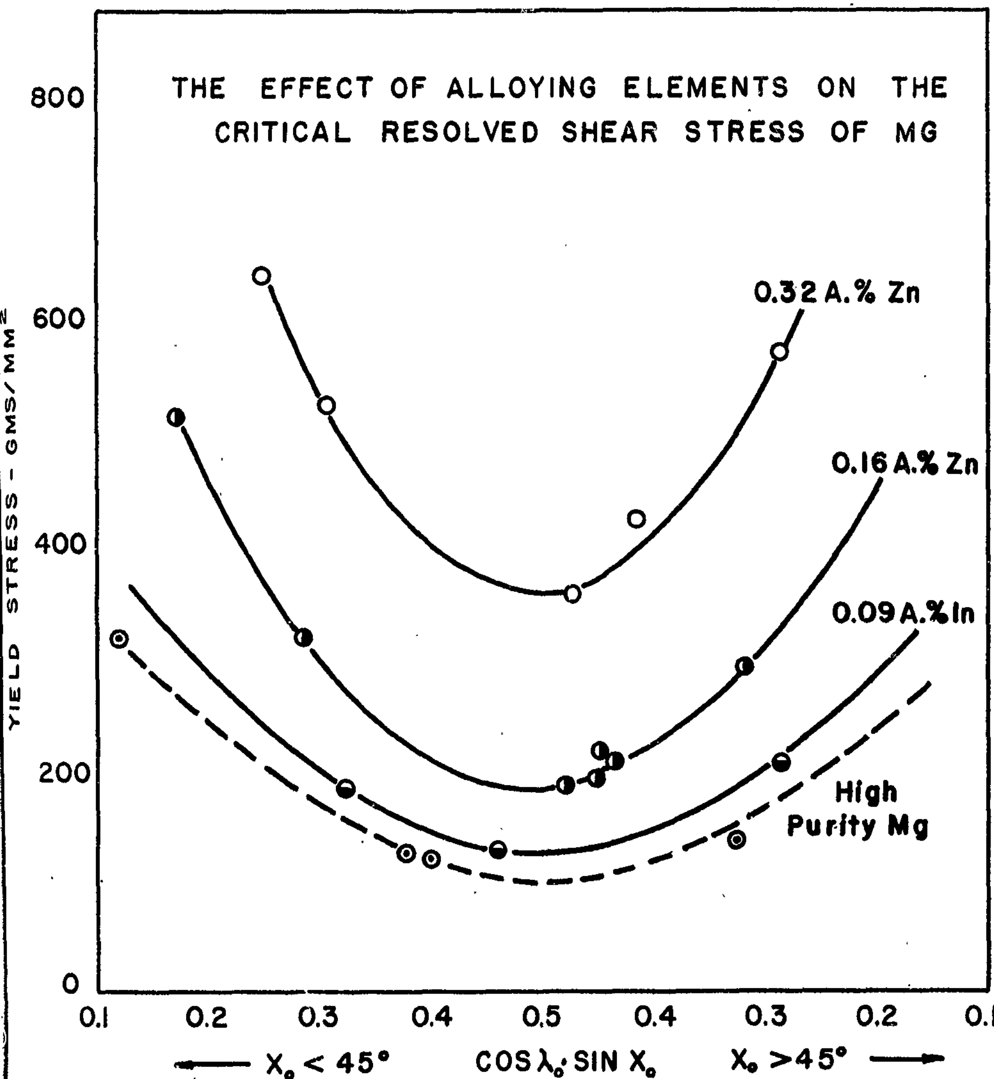
The excellent agreement between the present values and those previously reported for Mg attests to the validity of the testing procedure. Both sets of data gave an average value of 46 gm/mm^2 for the resolved shear stress of pure Mg.

For the Zn alloys tested, the critical resolved shearing stress increased almost linearly with increasing atomic per cent Zn. It was unfortunate that the Hg and Cd values had such erratic results since these alloys would have furnished resolved shear stress data for a constant e/a ratio. Furthermore, when reproducible values of the shear stress are obtained, the data will furnish an excellent test of the equation for the yield stress in solid solutions derived from dislocation theory by Mott and Nabarro.

REFERENCES

1. Doan, J., Pietrokowsky, P. and Tietz, T. Trans. AIME, 188 (1950) page 933.

2. Schmid, E. and Seliger, H. Metallwirtsch, 11 (1932) page 409.


THE EFFECT OF ALLOYING ELEMENTS ON THE CRITICAL RESOLVED SHEAR STRESS OF MG
0.32 A.% Zn
0.16 A.% Zn
0.09 A.% In
High Purity Mg
YIELD STRESS - GMS/MM²
800
600
400
200
0
0.1
0.2
0.3
0.4
0.5
0.4
0.3
0.2
0.1
← X₀ < 45°
COS λ₀· SIN X₀
X₀ >45° →

E.C.B.
4-30-53

TABLE I

ANALYSIS OF ALLOYS

Alloy No.	% Al	% Ca	% Cu	% Fe	% Mn	% Ni	% Pb	% Si	% Sn	% Zn	% Li	% Hg	% Cd
Sublimed Mg	<.0001	<.01	<0.0001	<0.0005	0.0014	<0.0002	0.0013	<0.001	<0.001	<0.02			
70405	0.36	<0.01	<0.0001	<0.0005	0.0014	<0.0002	0.0013	<0.001	<0.001	<0.02			
70406	1.70	<0.01	<0.0001	0.0006	0.0014	<0.0002	0.0013	<0.001	<0.001	<0.02			
70407	<0.002	<0.01	<0.0001	0.0006	0.0014	<0.0002	0.0013	<0.001	<0.001	0.42			
70408	<0.002	<0.01	<0.0001	0.0005	0.0014	<0.0002	0.0013	<0.001	<0.001	0.85			
70409	<0.002	<0.01	<0.0001	0.0006	0.0014	<0.0002	0.0013	<0.001	<0.001	<0.02	0.40		
70410	<0.002	<0.01	<0.0001	0.0006	0.0014	<0.0002	0.0013	<0.001	<0.001	<0.02	4.40		
73593	0.0001	<0.01	<0.0001	0.0007	0.0014	<0.0002	0.0013	<0.001	<0.001	<0.02		0.54	1.09
73594	0.00018	<0.01	<0.0001	0.0010	0.0014	<0.0002	0.0013	<0.001	<0.001	<0.02			5.03
73595	0.0001	<0.01	<0.0001	.0007	0.0014	<0.0002	0.0013	<0.001	<0.001	<0.02			11.43
73596	0.0001	<0.01	<0.0001	.0007	.0013	<0.0002	0.0013	<0.001	<0.001	<0.02			0.74
73597	0.0001	<0.01	<0.0001	.0007	.0013	<0.0002	0.0013	<0.001	<0.001	<0.02		1.28	
73598	0.0001	<0.01	<0.0001	.0006	.0013	<0.0002	0.0013	<0.001	<0.001	<0.02		3.82	
73599	0.0001	<0.01	<0.0001	.0004	.0013	<0.0002	0.0013	<0.001	<0.001	<0.02			

TABLE II

DATA OBTAINED FROM TENSILE TESTING OF SINGLE CRYSTALS

Crystal No.	Alloy	X_o*	λ_o*	cas $\lambda_o \sin X_o$	Yield Stress gm/~~min~~^{2} mm	Critical Resolved Shear Stress gm/~~min~~^{2} mm
70407-8	0.156 A.% Zn	51	54	0.456	184	84.0
-9	0.156 A.% Zn	10	10	0.171	513	87.6
-6	0.156 A.% Zn	52	52	0.485	180	87.8
-10	0.156 A.% Zn	50	55	0.439	210	92.0
-11	0.156 A.% Zn	55	56	0.452	215	97.4
-12	0.156 A.% Zn	70	70	0.322	280	90.0
-13	0.156 A.% Zn	17	17	0.280	314	88.1
70408-4	0.32 A.% Zn	45	48	0.473	343	162
-1	0.32 A.% Zn	35	35	0.470	347	163
-2	0.32 A.% Zn	57	60	0.420	418	175
-3	0.32 A.% Zn	15	15	0.250	640	160
-5	0.32 A.% Zn	72	72	0.284	564	160
-6	0.32 A.% Zn	19	19	0.306	523	161
71983-3	0.09 A.% In	36	39	0.456	126	57.4
-1	0.09 A.% In	72	72	0.284	172	51.2
-2	0.09 A.% In	20	20	0.322	183	59.0
24	Pure Mg	4	4	.127	384	48.7
21	Pure Mg	25	27	.376	122	45.8
8	Pure Mg	30	38	.394	112	44.2
7	Pure Mg	70	70	.321	147	47.1

* X_o = Angle between stress axis and basal plane.

* λ_o = Angle between stress axis and slip direction.